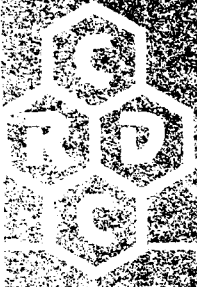END

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PHASE BEHAVIOR AND OILS IN POTASSIUM LINOLEATE/n-ALKANOL MICROEMULSIONS

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RESEARCH DIRECTORATE

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US Army Armament, Munitions & Chemical Command
Aberdeen Proving Ground, Maryland 21010-5423

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REPORT DOCUMENTATION PAGE

1a. REPORT SECURITY CLASSIFICATION UNCLASSIFIED		1b. RESTRICTIVE MARKINGS			
2a. SECURITY CLASSIFICATION AUTHORITY		3. DISTRIBUTION / AVAILABILITY OF REPORT Approved for public release; distribution unlimited.			
2b. DECLASSIFICATION / DOWNGRADING SCHEDULE					
4. PERFORMING ORGANIZATION REPORT NUMBER(S) CRDC-TR-84089		5. MONITORING ORGANIZATION REPORT NUMBER(S)			
6a. NAME OF PERFORMING ORGANIZATION CRDC	6b. OFFICE SYMBOL (If applicable) SMCCR-RSC-C	7a. NAME OF MONITORING ORGANIZATION			
6c. ADDRESS (City, State, and ZIP Code) Aberdeen Proving Ground, MD 21010-5423		7b. ADDRESS (City, State, and ZIP Code)			
8a. NAME OF FUNDING / SPONSORING ORGANIZATION CRDC	8b. OFFICE SYMBOL (If applicable) SMCCR-RSC-C	9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER			
8c. ADDRESS (City, State, and ZIP Code) Aberdeen Proving Ground, MD 21010-5423		10. SOURCE OF FUNDING NUMBERS			
		PROGRAM ELEMENT NO.	PROJECT NO. 1L161102	TASK NO. A71A	WORK UNIT ACCESSION NO.

11. TITLE (Include Security Classification)

Phase Behavior and QLS in Potassium Linoleate/n-Alkanol Microemulsions (U)

12. PERSONAL AUTHOR(S)
Mackay, Raymond A., and Seiders, Reginald, P.

13a. TYPE OF REPORT Technical	13b. TIME COVERED FROM 83 09 TO 84 01	14. DATE OF REPORT (Year, Month, Day) 1985 March	15. PAGE COUNT 19

16. SUPPLEMENTARY NOTATION

17	COSATI CODES		18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)		
FIELD	GROUP	SUB-GROUP	Phase behavior		Butanol
15	02		Quasielastic light scattering		Pentanol
			Microemulsions		Hexanol

19. ABSTRACT (Continue on reverse if necessary and identify by block number)

We present here pseudo-ternary phase maps of three surfactant stabilized quaternary systems composed of water, hexadecane, and potassium linoleate (KL) as surfactant and the three saturated primary alcohols, 1-butanol, 1-pentanol, and 1-hexanol as cosurfactants. These systems exhibit phase behavior analogous to that of the potassium oleate/n-alkanol systems recently reported. Butanol gives a system with one large single-phase region extending from the water side to the oil side of the diagram. Pentanol yields two areas of single phase, a large upper water in oil (w/o) region and a smaller finger-like oil in water (o/w) region arcing up from the water apex to ca 20% emulsifier. The hexanol system reveals the largest expanse of the w/o region, but in the water-continuous corner only a metastable clear area was encountered. Quasielastic Light Scattering (QLS) measurements are also reported in all three systems, and an attempt is made to correlate these measurements to the phase behavior. The butanol system gave no QLS evidence of aggregation at three different compositions. The

20. DISTRIBUTION / AVAILABILITY OF ABSTRACT [X] UNCLASSIFIED/UNLIMITED ☐ SAME AS RPT. ☐ DTIC USERS	21. ABSTRACT SECURITY CLASSIFICATION UNCLASSIFIED	
22a. NAME OF RESPONSIBLE INDIVIDUAL BRENDA C. ECKSTEIN	22b. TELEPHONE (Include Area Code) (301) 671-2914	22c. OFFICE SYMBOL SMCCR-SPS-IR

DD FORM 1473, 84 MAR   83 APR edition may be used until exhausted.   SECURITY CLASSIFICATION OF THIS PAGE
All other editions are obsolete.

18. Subject Terms (continued)

Pseudo-ternary
Potassium linoleate/n-alkanol

19. Abstract (continued)

pentanol and hexanol system gave diffusion coefficients that suggest the existence of aggregates with correlation lengths in the 100- to 700-Å range. In general, diffusion coefficients for the hexanol system were smaller than those at comparable compositions in the pentanol system. In all three systems at low water content (<20%) in the single-phase region near the E-O axis, QLS gives no evidence of aggregation. Finally, we report that diffusion coefficient measurements at compositions near phase boundaries in the pentanol and hexanol systems gave low values that were interpreted as manifestations of proximity to critical points.

PREFACE

The work described in this report was authorized under Project No. 1L161102A71A, CB Defense Research. This work was started in September 1983 and completed in January 1984.

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CONTENTS

LIST OF FIGURES

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PHASE BEHAVIOR AND QLS IN POTASSIUM LINOLEATE/n-ALKANOL MICROEMULSIONS

1.   INTRODUCTION

Microemulsions have recently attracted considerable interest by virtue of their diverse uses, particularly in tertiary oil recovery.[1,2] The so-called bicontinuous[3] or cosolubilized[4] systems in particular have received much attention because of the internal structural changes that must occur, but are poorly understood, as the clear region is traversed in pseudo-ternary phase maps of these microemulsions. "Abnormal" microemulsions, which exhibit only a single clear region in their pseudo-ternary phase maps, are stabliized by consurfactants that are typically short-chain linear alcohols.[5] Cetyltri-methylammonium bromide (CTAB)/n-butanol/hexadecane[6] and sodium dodecylsufate/butanol/toluene[7] are examples of such systems. It has been repeatedly shown that various physicochemical properties of microemulsions are strongly affected by the choice of cosurfactant. Shah[4] had used conductometry, viscosimetry, NMR, x-ray scattering, and electron microscopy to conclude that substitution of 1-pentanol for 1-hexanol in water/potassium oleate/alcohol/hexadecane systems results in the formation of a cosolubilized system (molecular solution) from what had been a true microemulsion. Clausse[8] reached the same conclusion through electrical conductivity studies, and suggested that, in general, micro-emulsions and cosolubilized systems could be differentiated by determining whether they fit the Percolation and Effective Medium theories.[9,10]

In contrast, Mackay et al. have shown that in microemulsion systems utilizing butanol or pentanol as cosurfactants, there appear to exist discreet hydrophilic and hydrophobic regions.[11] By polarographically measuring diffusion coefficients of electroactive oil soluble probes in CTAB/butanol and SCS/pentanol systems, it was shown that the probe does not diffuse freely. Rather, it has the smaller diffusion coefficient that one would expect if it were associated with hydrophobic aggregates. The results from using this polarographic technique in other microemulsions and in micelles have been shown to compare favorably with diffusion coefficient values obtained by quasielastic light scattering on identical systems.

In order to further elucidate the role of alcohol cosurfactant and surfactant polyunsaturation on microemulsion behavior, we report here the determination of pseudo-ternary phase maps of hexadecane/water systems stabilized by KL/1-alkanol emulsifier, and the examination of the single-phase regions by means of QLS. Our primary interest in choosing linoleic acid is to investigate its polymerization in microemulsions after the systems are adequately character-ized.

2.   EXPERIMENTAL

2.1   Materials.

Linoleic acid (Sigma, 99% or Fisher reagent grade), 1-butanol and 1-pentanol (B&A) and 1-hexanol (Eastman) were used as received. Proton NMR and microemulsion phase behavior for the linoleic acid were independent of its source. Hexadecane (Humphrey Cml Co) was dried over 4-A sieves before use. Potassium hydroxide (87%, solid) was obtained from Fisher (certified A.C.S. grade).

The emulsifier E for each phase map was prepared by dissolution of KOH (1.05 eq) in a solution of linoleic acid (1 eq) and the appropriate alcohol (6 eq) with stirring and/or gentle heating. The reported water content of compositions in the phase maps are not corrected for the small amount of water present from the saponification of the acid. The phase maps were prepared in the usual manner of starting at several compositions on an axis (E-W or E-O) and titrating to clear or turbid endpoints with the final component (oil or water).

2.2   QLS Measurements.

A fixed-angle (90°), laser light scattering photometer (Model LSA-1) from Langley-Ford Instruments was used. The LSA-1 was modified with a Spectra-Physics 15mW 632.8-nm HeNe laser source and contained a scattered light-collection aperture that defined approximately one coherence area. The autocorrelation functions were computed with Langley-Ford (Model 1096) computing correlator. The analysis program employs the method of cumulants[12] to fit the correlation function to a single exponential decay directly in the correlator itself, without the use of a separate external computer. Calculations are then performed using the results of the cumulants analysis to yield the average translational diffusion coefficient and subsequently, from the Stokes-Einstein equation, the corresponding correlation length. The continuous phase was assumed to have the viscosity and refractive index of pure water at 25°C unless otherwise indicated. Samples were filtered through a 0.45-um filter directly into the cuvette to minimize dust incorporation. The samples were then maintained at $25° \pm 0.1°C$, unless otherwise stated by means of water circulation in the cuvette cell block from a constant temperature bath. The titration to give droplet size as a function of water dilution was done directly in the cuvette by addition of known aliquots of water from a microliter syringe to the filtered E-O solution. The stoppered cuvette was then shaken to mix (endothermic heat of mixing was noted) and equilibrated in the cell block for 10 min; data was then collected. In all systems investigated, the mole ratio of cosurfactant alcohol to surfactant was maintained at six. This corresponds to a mass ratio of 1.4:1 (butanol:KL) to 1.9:1 (hexanol:KL).

3.   RESULTS AND DISCUSSION

3.1   Phase Maps.

Figures 1 through 3 show the psuedo-ternary phase maps of the three systems investigated: hexadecane/water/KL/butanol, pentanol, or hexanol. The general trend observed with increasing alcohol chain length is analogous to that which has been observed in other systems.

Butanol, the most water soluble alcohol of the group, gives a clear region that extends from near the water (W) to near the hexadecane oil (O) apex. This behavior is reminiscent of the water/SDS/butanol/dodecane system recently reported by Clausse et al.[8] It is also similar to the behavior reported for the water/potassium oleate/pentanol/hexadecane system that has been so thoroughly studied by Shah[4] and coworkers. These latter two systems have been described as cosolubilized systems or molecular solutions. The similarity of the butanol system here to those systems suggests that it too might be of the cosolubilized type. Our QLS measurements of this system are not inconsistent with this view. Measurements of diffusion coefficients at the three compositions

8

labelled A, B, and C in the clear region of this system (Figure 1) failed to give an indication of droplets in the solution. The QLS data consisted of the type of scattering found in pure solvents or molecular solutions and thus would suggest that there are no distinct hydrophilic and hydrophobic regions in this system. Two other interpretations are also possible. As in any light scattering experiment a matching of refractive indices of the aggregate and the continuous phase would prevent detection. We find this interpretation unlikely in light of the fact that we do see aggregates when either pentanol or hexanol are used instead of butanol, and the refractive indices of the alcohols are not greatly different. The second interpretation of the QLS data could be that there are rapidly intercoverting small aggregates such as suggested by Scriven[3] that complicate the interpretation. We can not rule out this interpretation, and recent polarographic diffusion measurements of Mackay[11] suggest that discrete hydrophobic regions do exist in similar systems for which QLS measurements give unreasonably small effective diameters.

For the pentanol system (Figure 2) we find two clear phases such as were found in the water/potassium oleate/hexanol/hexadecane system studied by Shah et al.[4] and Clausse et al.[8] These systems consist of two clear regions that are separated by a viscous, turbid, birefringent zone. In the linoleate system, the larger upper region, with generally less than 50% water, is identified as a water-in-oil (w/o) type region based on its connection with the O apex, while the lower fingerlike region, arching up from the W apex contains less than 20% oil and should be of the oil-in-water (o/w) type. The transition from w/o to o/w systems that gives rise to such striking changes in macroscopic properties is also reflected in the QLS measurements, as will be discussed below.

The hexanol system (Figure 3) again shows a large upper w/o region that is even larger then in the pentanol system. While there may be also a small o/w region near the W apex, it is our belief that it is most likely a metastable region, and it was difficult to reproduce on repetitive titrations. Thus it appears that although hexanol stabilizes w/o microemulsions more effectively than pentanol, the roles are reversed for stabilization of o/w microemulsions in the present system. Furthermore, from comparison of the clear areas in the three figures, it appears that hexanol is the most efficient cosurfactant in the mutual solubilization of water and hexadecane in the presence of potassium linoleate.

3.2   QLS Measurements.

For the QLS measurements, monodisperse particles give rise to an autocorrelation function that decays exponentially with a 1/e decay time $\tau = \Gamma^{-1}$. The diffusion coefficient (D_z) of the particle is given by ΓK^{-2}, where $K = (4\pi n/\lambda) \sin \theta/2$. Here λ, θ, and n are the laser wavelength (632.8 nm), scattering angle (90°), and refractive index of the continuous medium, respectively. If spherical geometry is assumed, the hydrodynamic diameter d_z can be calculated from the well known Stokes-Einstein equation $d_z = kT/3\pi n D_z$, where n is the viscosity of the continuous medium, k is Boltzman's constant, and T is the absolute temperature. For a polydisperse distribution of particles, D_z is the Z-average diffusion coefficient and is weighted in favor of the larger particles. The variance obtained from the QLS data is a measure of the polydispersity. As a rule of thumb, variances less than 0.1 will be considered to be reasonably monodisperse.
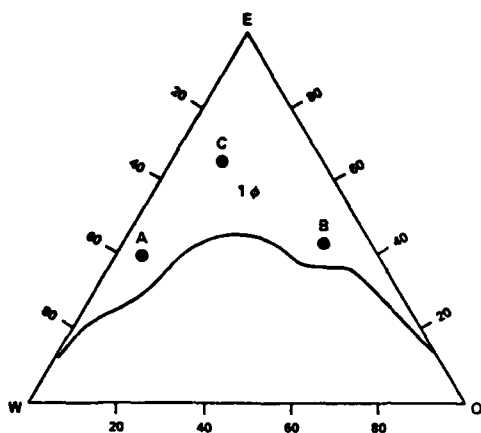
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Figure 1.   Psuedo-Ternary Phase Map for the Water/KL/1-Butanol/Hexadecane System

The emulsifier E consists of a 1:6 mole ratio of surfactant to cosurfactant.
QLS measurements were done at compositions labelled A, B, and C in the clear
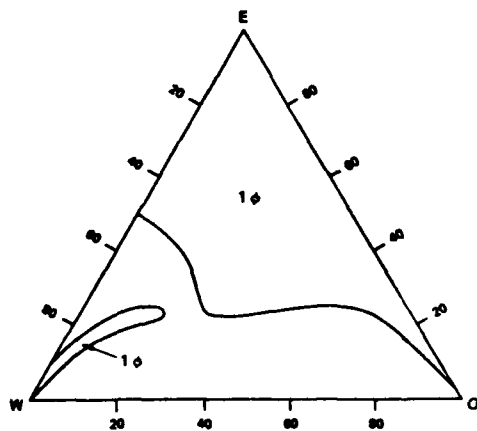region.

Figure 2:   Pseudo-Ternary Phase Map for the Water/KL/1-Pentanol/Hexadecane System

The emulsifier E consists of a 1:6 mole ratio of surfactant of cosurfactant.
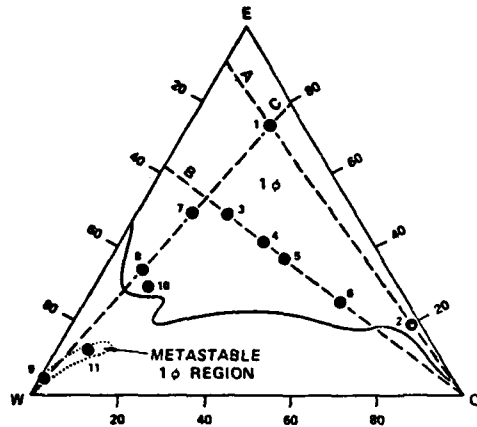See Table 1 for diffusion measurement data.

10

Figure 3: Pseudo-Ternary Phase Map for the Water/KL/1-Hexanol/Hexadecane System.

The emulsifier E consists of a 1:6 mole ratio of surfactant to cosurfactant.
See Table 2 for diffusion measurements along dilution line A, B, and C.

We have used QLS to measure diffusion coefficients (D_z) in each system described above. As stated earlier, the butanol system gives scattering similar to that of a molecular solution or of a surfactant solution below the critical micelle concentration. No decay curve was observed in the autocorrelation function. In contrast, the pentanol (Table 1) and hexanol (Table 2) systems were rich in particles whose D_z's varied over a broad range. Figure 4 shows the variation in apparent size, assuming spherical droplets (using the refractive index and viscosity of pure water) as a function of water content along the dilution line running from 60 wt % E/40 wt % O to the water apex. In general, the sizes correlate well with phase behavior in that large changes are observed near the phase boundaries; however, it should also be noted that the effective diameters (correlation lengths) reported include the effect of interactions and near demixing lines, concentration fluctuations. Therefore the numbers quoted do not necessarily reflect true particle sizes.

At some points taken in other systems where the solubilized phase is high, a correlation between the diffusion coefficient and size has been made.[13] From 0% to 20% water, no droplets are detectable by QLS. Similar results are obtained in the butanol and hexanol systems. From ca 22% to 45% water, droplets of ca 90-A effective diameter are seen. This behavior agrees very nicely with that reported b,   ˜rg[14] for the water/potassium oleate/pentanol/decane system. He reported that no   ‹lets were detected until the water concentration was increased to the 20% . ˀ5% range depending on alcohol to soap ratio. It was suggested that this col ntration of water was necessary in order to fully hydrate each soap moler e headqroup and counterion. Scattering intensity would increase and th‹ ‹roplets would form only at water contents above this 20%-25% range. Our ol ervations are in agreement and suggest that the additional unsaturation in the fa ty acid in our system does not affect the basic aggregation characteristics in this w/o region. Their population appears to increase with water content in this region as evidenced by the diminished time that is required to obtain a suitable smooth decay function (e.g., 1-min collection time at 40%

11

Table 1. Summary of QLS Results on KL/Pentanol/C_{16} System Along
Dilution Line from 40% O/60% E

% Water	D^a	ξ^b (Å)
10	0^c	-
20	0^c	-
25	5.74	86
28	5.17	95
31	5.50	89
36	5.71	86
40	5.45	90
44	5.91	83
47	4.24	117
48.4	1.92	257
50	3.52	141
52	2.32	211
58	5.33	92
59	4.42	112
63	2.10	224
66	0.91	560
68.3	0.21	1950

[a]Apparent diffusion coefficient (x 10^7 cm^2 S^{-1}) calculated using n_d = 1.33

[b]Correlation length, calculated from the Stokes-Eistein equation, using 25° and viscosity of 0.089 poise

[c]Correlation function was flat

12

Table 2. Summary of QLS Results on KL/Hexanol/C_{16} System

Composition[a]	D_z	ξ[b] (A)
1	0[c]	-
2	0[c]	-
3	0.741	662
4	1.11	441
5	1.19	344
6	1.53	320
7	1.28	382
8	0.734	668
9	2.09	235
10	0.175	2800
10	0.233	2100
11	0.233	2100

[a]From KL/hexanol phase map

[b]Correlation length, calculated from the Stokes-Einstein equation, using a temperature of 25°C and viscosity of 0.089 poise

[c]A flat correlation function was obtained.
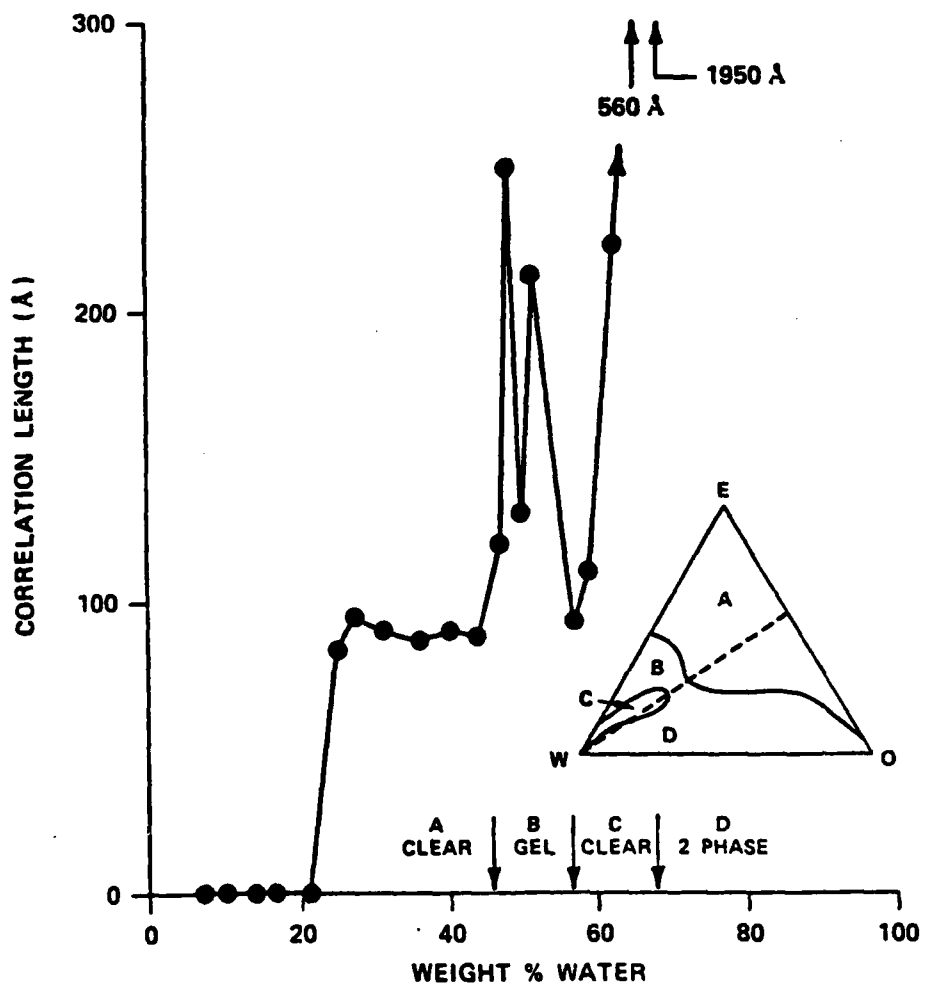
[d]$\times 10^7$ cm^2/s. Calculated using n_d = 1.33.

Figure 4: Correlation Length from QLS (Using n = 0.89 cp, n_d = 1.33) Along
the Dilution Line from 60% E/40% O to Water Apex (dashed line in insert)
for the KL/1-Pentanol/Hexadecane System

vs 5 min at ca 25% water). At the phase boundary between the w/o microemulsion region (A) and the gel region (B), the correlation length increases dramatically as one might expect from the large aggregates that must make up the liquid crystals in this region. The greatly increased viscosity in this birefringent gel region also suggests extended aggregate formation here. Continuing on the dilution line through the B area reveals large fluctuations in correlation length, and at the boundary to the o/w region (C) the length drops back down to ca 100 A at 57% water, which is just inside the clear w/o region. The correlation length then increases sharply as the next phase boundary is approached at ca 68% water and region D is entered. Thus, these length increases which appear to correlate with the macroscopic phase boundaries appear to be evidence of approach toward critical points in this system.[15,16]

Finally, in the pentanol system, we prepared the ternary phase map (Figure 5) of water/pentanol/potassium linoleate in order to determine the phase behavior at 0% oil. The dotted line from point E to the water apex is the same E-W axis that appears in Figure 2 for the pentanol E-O-W pseudo-ternary phase map, where E is a 1:6 mole ratio of surfactant to cosurfactant. Note that the compositions in Figure 5 are weight percents. The gross features of this figure, again, are two clear regions separated by a turbid viscous gel zone. This behavior suggests that these phases are the origins of the clear regions when oil is added to the system. The behavior is expected in light of Friberg's[14] thorough work which shows that the solubility areas in the quaternary systems can be traced back to 0% oil, ternary systems. However, additional "slices" parallel to the W-S-A plane are needed to definitely prove this point since, in some instances, the clear region on the S-A axis can contribute to the "o/w" region of its pseudo-ternary E-S-W phase map. No QLS measurements were carried out in the ternary system.
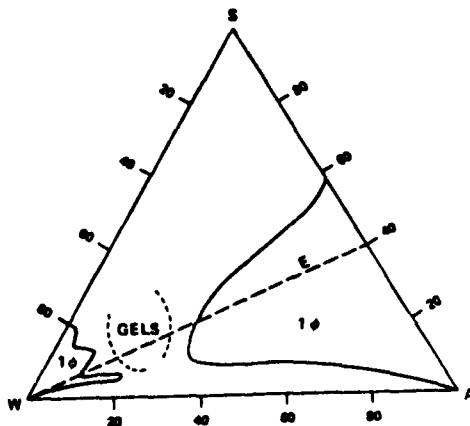

Figure 5: Ternary Phase Map of Water/KL/1-Pentanol

The dotted line (E) corresponds to the ratio of KL to pentanol used in Figure 2. Water (W) plus 1-pentanol (A) plus potassium linoleate (S) = 100 wt %.

15

As stated earlier, several diffusion coefficient measurements were also carried out in the hexanol system along three dilution lines, A (from 10% W, 90% E), B (from 40% W, 60% E), C (from 20% O, 80% E), and at several additional points as indicated in Figure 3. The diffusion coefficients (Table 2) are in general smaller than were found in the pentanol system (Table 1), suggesting somewhat larger aggregates due to both the increased size of this cosurfactant and its increased oil solubility over the pentanol. The sharp fluctuations in D (Table 2 and Figure 3) near boundaries (or metastable regions), points 8-11, are again observed as in the pentanol system. Points 1 and 2 with 10% water, which may be insufficient to form aggregates, give only flat autocorrelation functions characteristic of molecular solutions. Along the B dilution line, points 3-6, a gradual increase in D is observed with increasing oil content, suggesting breakup of larger aggregation or, perhaps more reasonably, dilution of the aggregates and less interparticle interaction, as has been suggested as the primary and expected influence of hydrocarbons.[14] Further along the water dilution line at points 7 and 8, a slight decrease in D is seen in the w/o region, but D increases again in the metastable region at very high water content (point 9). Points 10 and 11 are very near boundaries of the w/o and o/w areas, respectively, and show very small values relative to the other values in the system. The composition corresponding to 10 was prepared twice and gave quite different values for D (1.75 vs 2.33×10^{-8} cm^2 S^{-1}). We suggest that this is because of the closeness of this point to the boundary. In general, then, for this phase map (Figure 3), if one stays away from the phase boundary of the w/o region, values of D are obtained that are consistent with the view that aggregates are present. The metastable (20-min equilibration) nature of the high-water content compositions in the expected o/w region preclude reasonable discussion of this area.

4.   SUMMARY

We have presented pseudo-ternary phase maps of three new surfactant stabilized quaternary systems involving water, hexadecane, potassium linoleate as surfactant, and the three saturated primary alcohols (butanol, pentanol, hexanol) as cosurfactants. These systems were shown to exhibit phase behaviors similar to those of the potassium oleate/n-alkanol systems studied by Shah[4] and Clausse.[8] The shortest alcohol, butanol, gives a system with one large single-phase region extending from the water side to the oil side of the diagram. Pentanol gives rise to two areas of single phase behavior: a larger upper w/o region and a finger-like o/w region arcing up from the water apex to ca 20% oil and ca 25% emulsifier. The hexanol system contains the largest expanse of the w/o region but, in the water continuous corner, only a metastable clear area was encountered.

QLS diffusion measurements have also been reported in all three systems. The butanol system gives no evidence of aggregate existence, suggesting that cosolubilization may be occurring here. The pentanol and hexanol systems, however, give diffusion coefficients that clearly reveal aggregates with correlation lengths in the 100- to 700-A range. In general the diffusion coefficients for the hexanol system are smaller than those at comparable compositions in the pentanol system. In all three systems, at low water content (<20%) in the single-phase region near the E-O axis, QLS gives no evidence of aggregate formation. This is in line with the results of Friberg[14] on a similar system in which he concluded that a minimum ratio (8) of water-to-soap molecules is needed before reverse micelles are formed and detected in this region.

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Finally, diffusion coefficient measurements at compositions that correspond to proximity to phase boundaries in the pentanol and hexanol systems gave very low values. This was interpreted as possible evidence of proximity to critical points.

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